

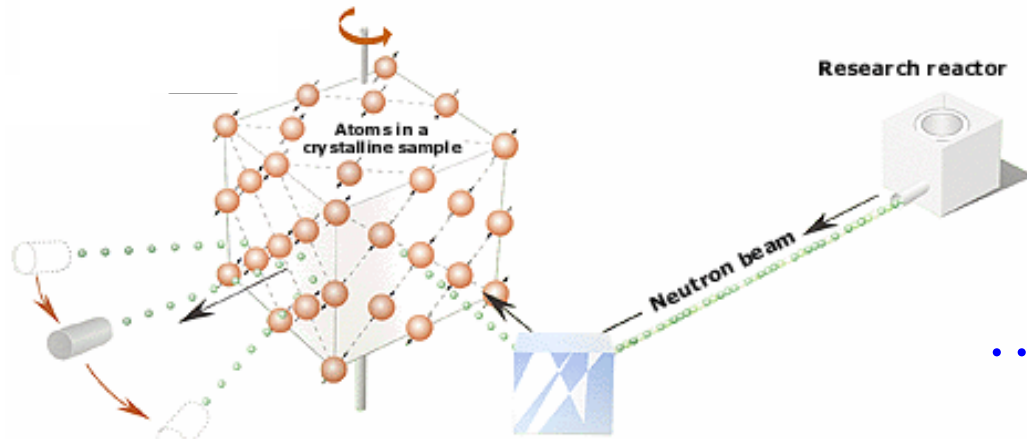
# Advanced Instrumentation for Neutron Scattering Studies of Nano-Scale Materials

Roger Pynn

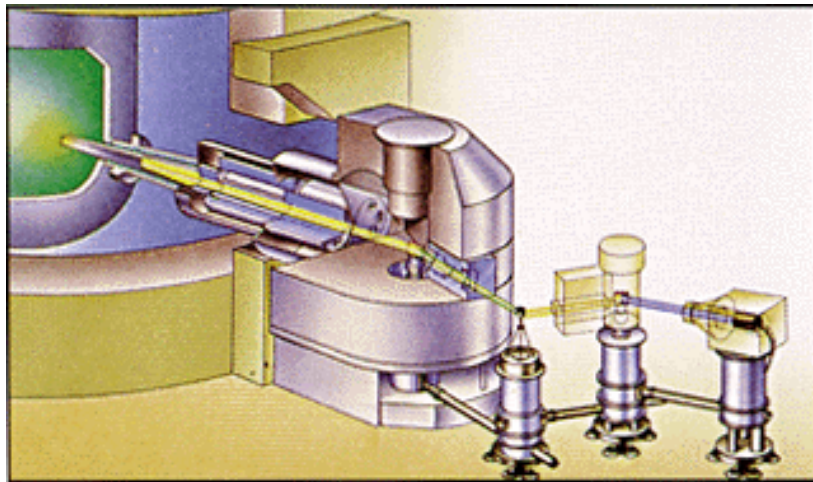
Los Alamos National Laboratory

# The 1994 Nobel Prize in Physics – Shull & Brockhouse

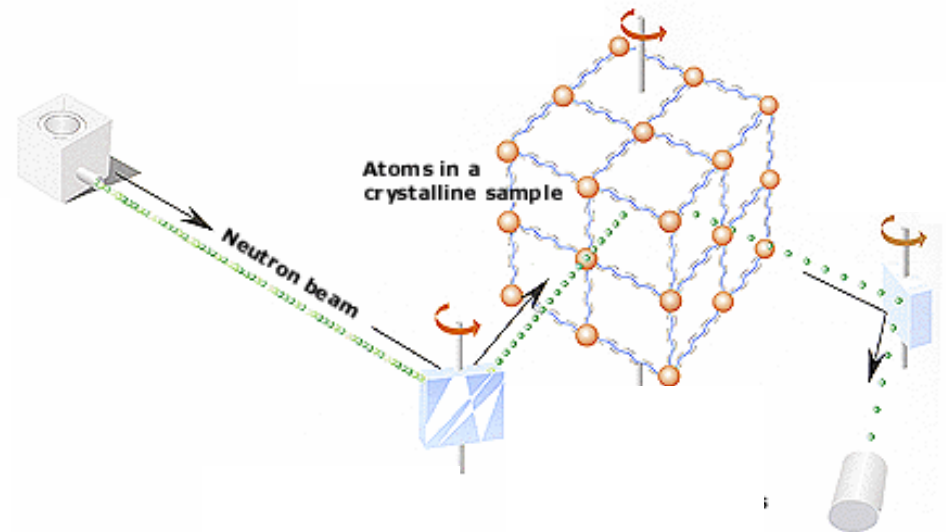
Neutrons show where the atoms are....



...and what the atoms do.



3-axis spectrometer

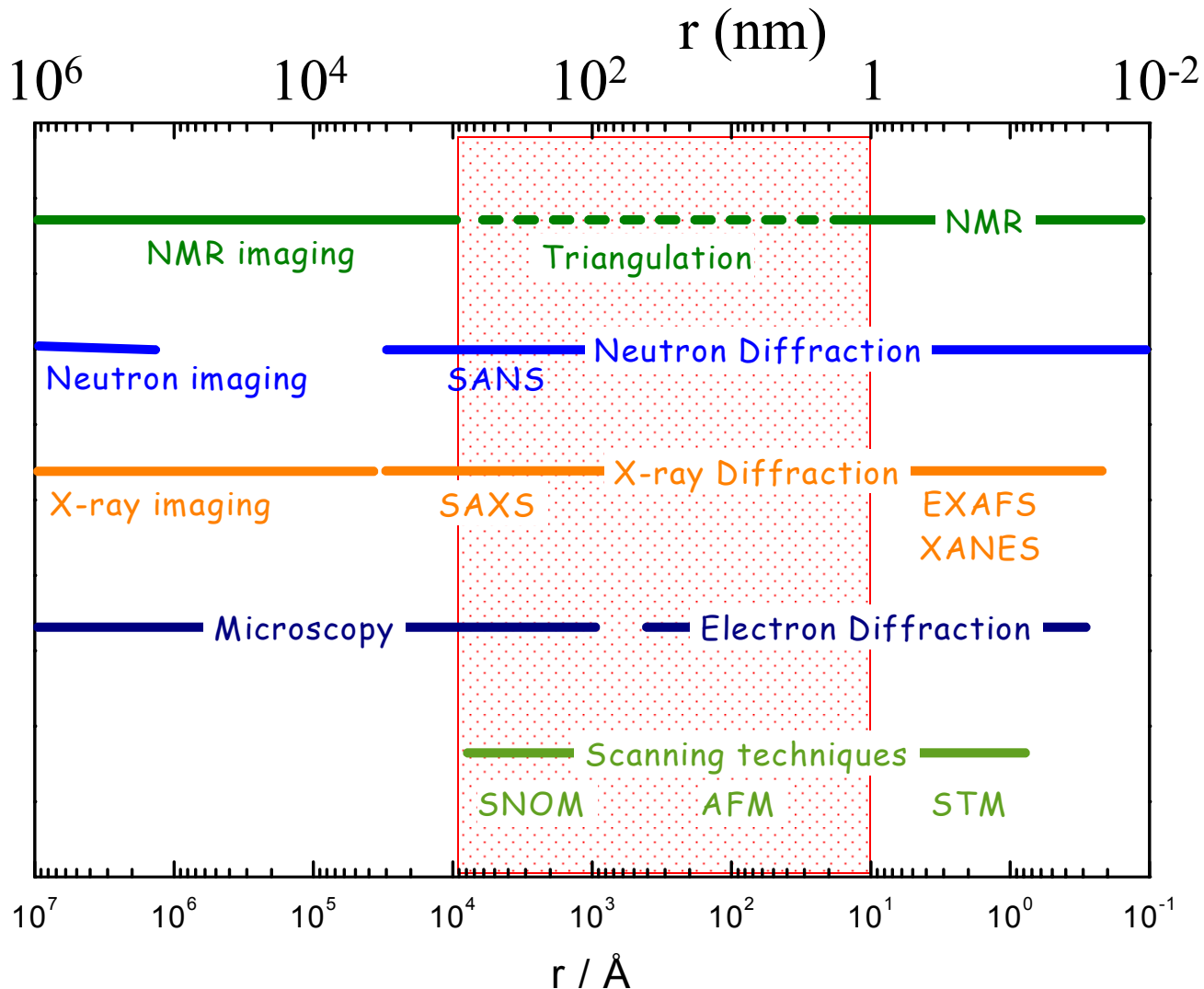


# The Success of Neutron Scattering is Rooted in the Neutron's Interactions with Matter

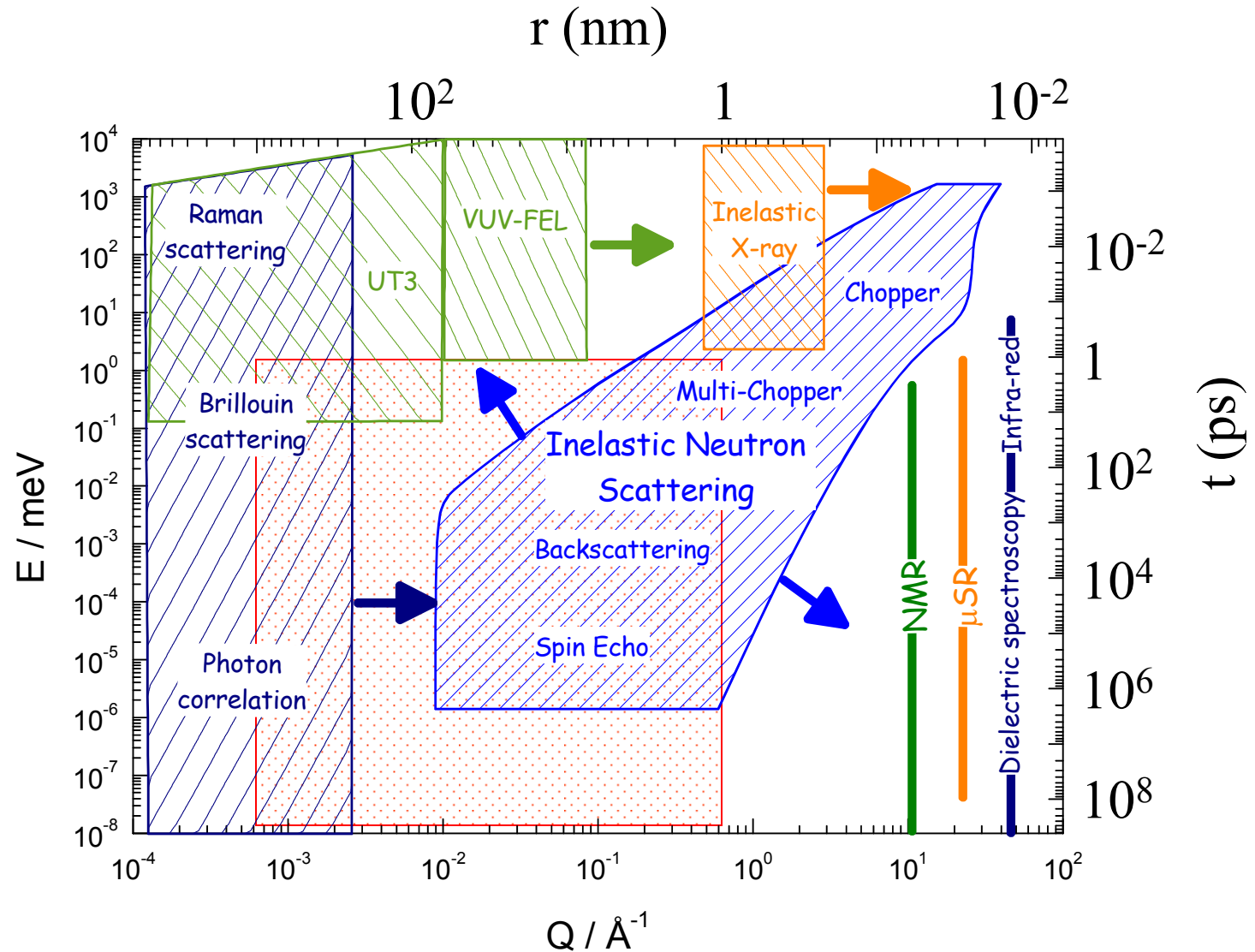
- Nuclear and magnetic interactions of similar strength
- Isotopic sensitivity (especially D and H)
- Penetrates sample containment
- Sensitive to bulk and buried structure
- Simple interpretation – provides statistical averages, not single instances
- Wavelength similar to inter-atomic spacings
- Energy similar to thermal energies in matter

**All of these apply to studies of nano-materials**

# Neutron Scattering Complements Other Techniques in Length Scale....



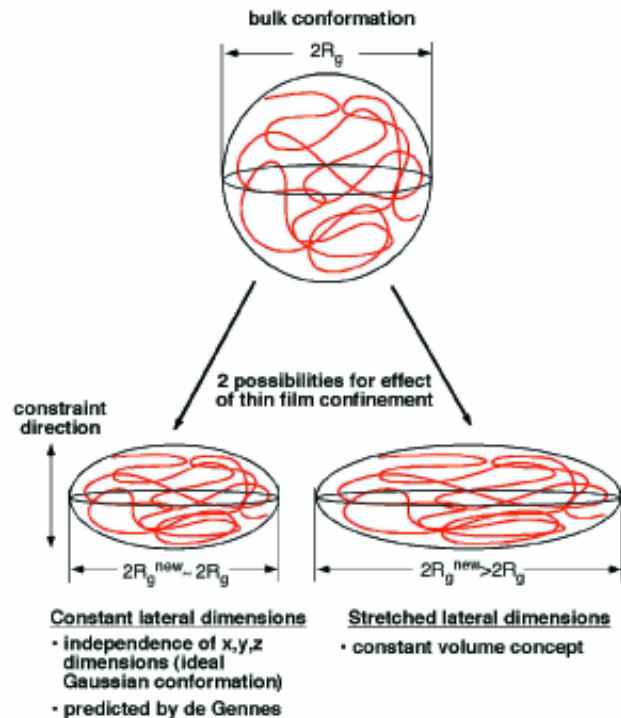
.....and Time Scale



# Neutron Scattering has Helped Resolve Structural Issues in Nano-Materials

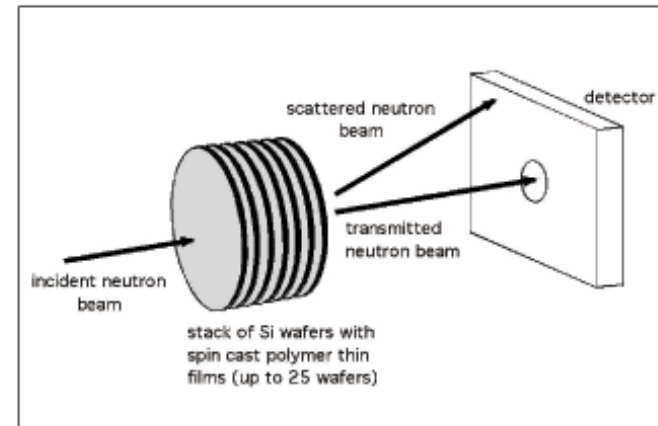
- Microstructure in bulk & thin films of complex fluids

# Probing Chain Conformation in Thin Films

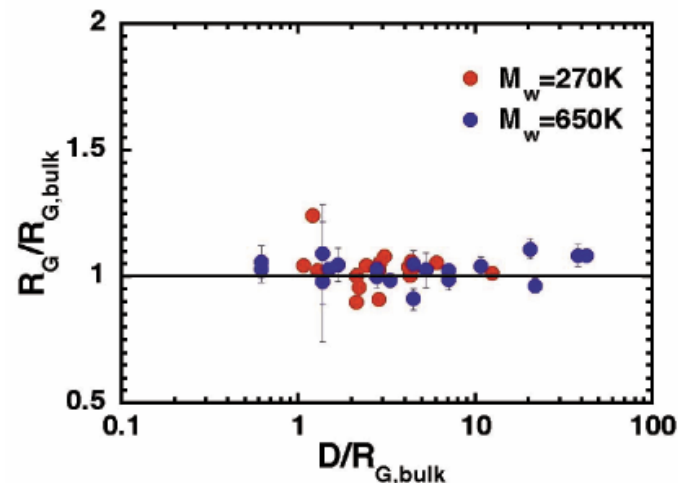


R.L. Jones, S.K. Kumar, D.L. Ho, R.M. Briber, T.P. Russell,  
*Nature*, 1999, 400, 146

Shows a limitation of neutron scattering for nano-science:  
 normally need large samples



Thin films of 25% d-PS & 75% PS spun on to Si wafers. 25 wafers  $\Rightarrow$  250 nm or larger total polymer thickness



$R_g$  in the plane of the film is unchanged down to film thickness of  $R_g/2$

# Neutron Scattering has Helped Resolve Structural Issues in Nano-Materials

- Microstructure in bulk & thin films of complex fluids
- Magnetic structures of many thin film systems
  - Proximity effects related to the importance of interfaces

# New challenges for magnetic neutron scattering

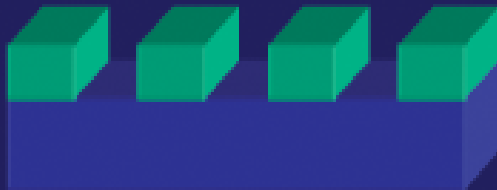
Spin valves



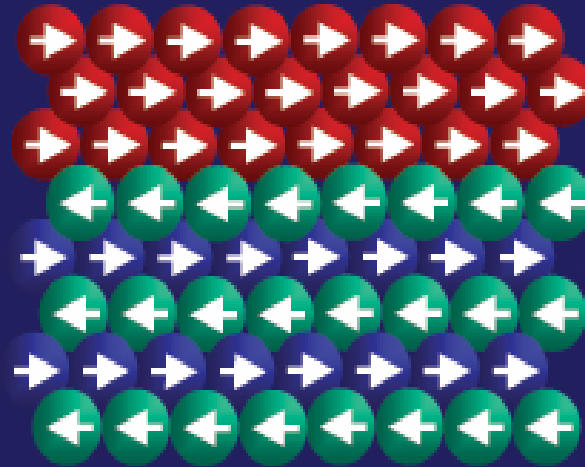
FM – Semiconductor



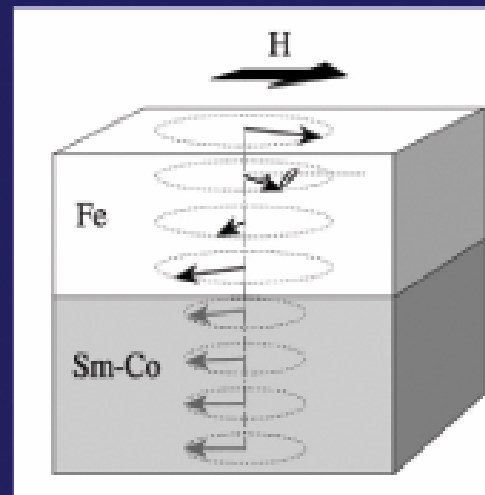
Lateral structures



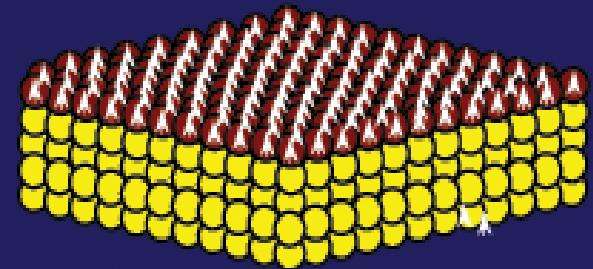
Exchange bias



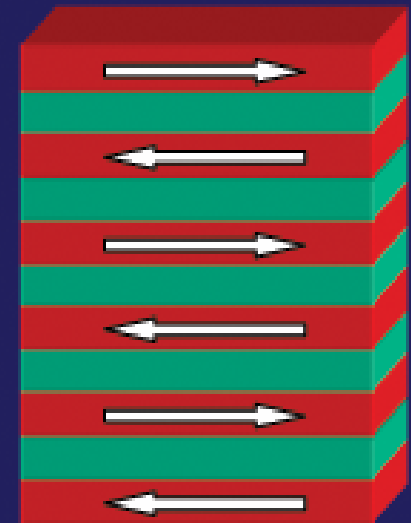
Exchange springs



Magnetic films,  
dimensionality effects

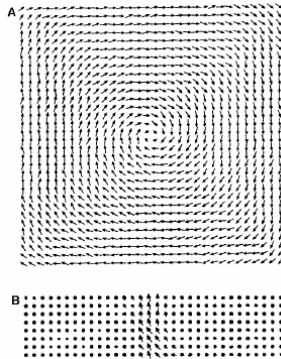
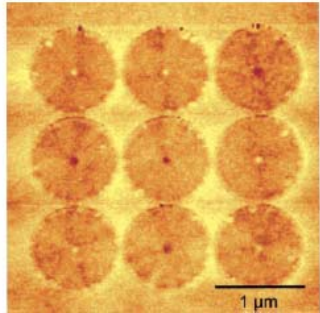


Exchange  
coupling

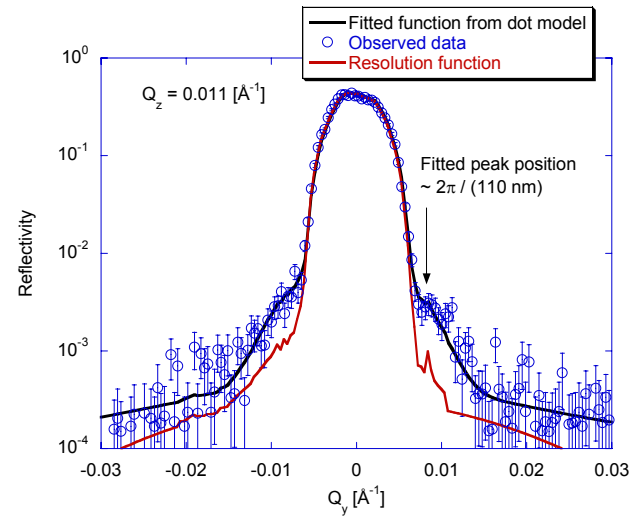


# Vortex State in Thin Films of Magnetic Dots

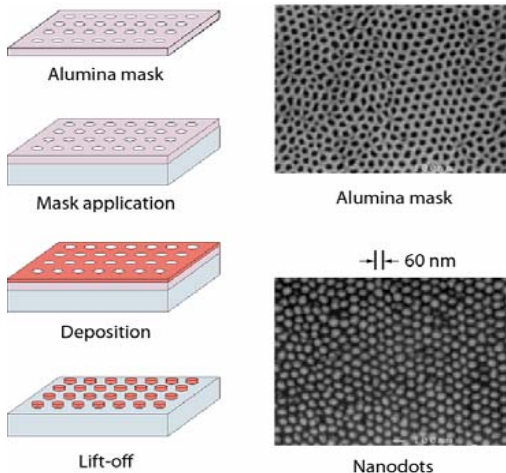
Shinjo *et al.*,  
Science **289**, 930 (2000)



Large ( $\sim 1 \mu\text{m}$ ) magnetic dots (above) are visible with MFM or neutron reflection. Small ( $\sim 65 \text{ nm}$ ) dots are harder to see



**65 nm diameter dots spaced  $\sim 110 \text{ nm}$  apart**

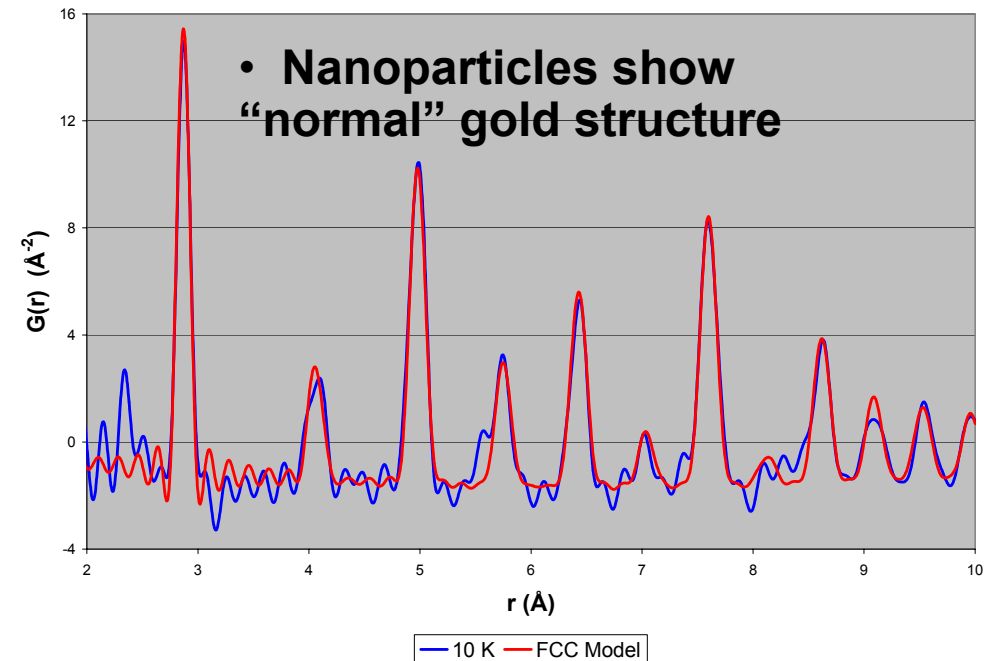
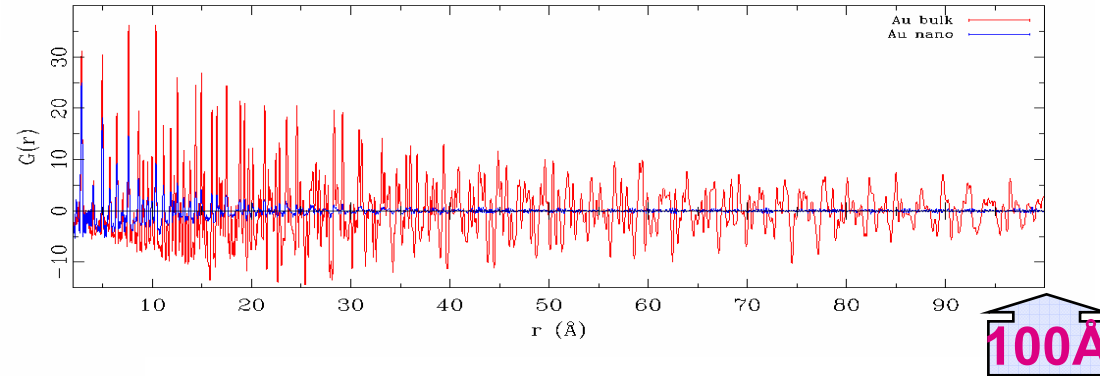
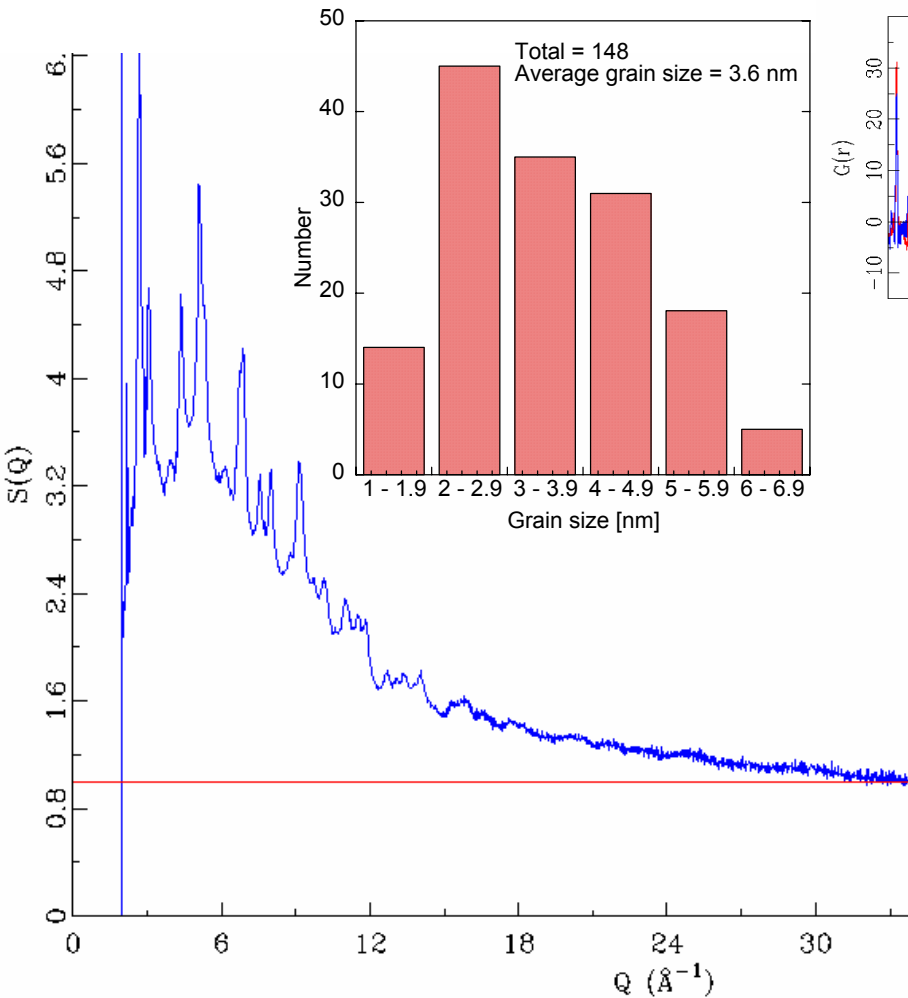


- GINS experiment with polarized neutrons
- Determined total moment in vortex state in each dot
- At the limit of today's neutron technology

# Neutron Scattering has Helped Resolve Structural Issues in Nano-Materials

- Microstructure in bulk & thin films of complex fluids
- Magnetic structures of many thin films
  - Proximity effects related to the importance of interfaces
- Atomic arrangements in nano-particles

# Neutron PDF Shows that Gold Nano-Particles Appear to have the Bulk Structure



# Neutron Scattering has Helped Resolve Structural Issues in Nano-Materials

- Microstructure in bulk & thin films of complex fluids
- Magnetic structures of many thin films
  - Proximity effects related to the importance of interfaces
- Atomic arrangements in nano-particles
- Elastic constants of micro-emulsion droplets (NSE)
- Dynamics of stacked membranes (TAS)
- etc

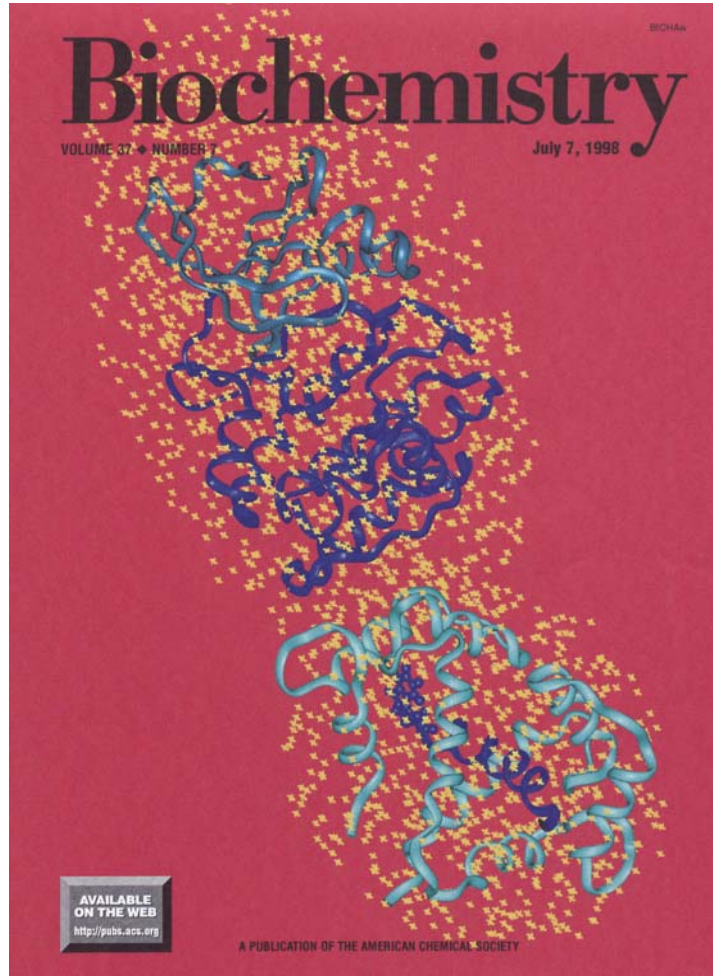
# What do we Need to do Better?

- Exploit complementarity of techniques
- Generate pictures not  $S(Q,E)$ 
  - Couple neutron scattering and advanced computing
  - Prototypes exist for powder diffraction, SANS and quasielastic scattering

# Integration of Structural Biology Tools Yields Insight into Enzyme Activation by Calmodulin

**Crystallography** – structure of the catalytic core of the enzyme and reveals the location of the **catalytic cleft**.

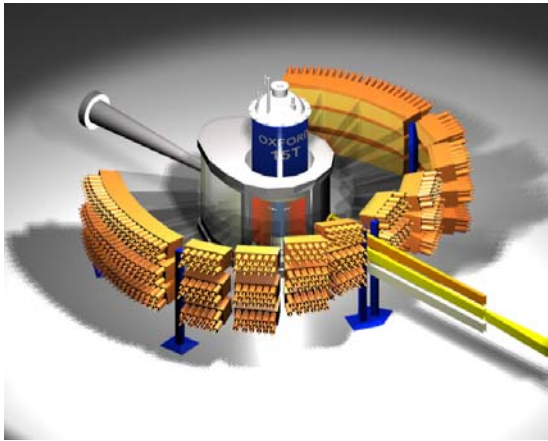
**High field NMR with isotope labeling** – high resolution solution structure of calmodulin complexed with its **binding domain** from the enzyme.



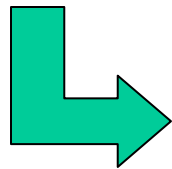
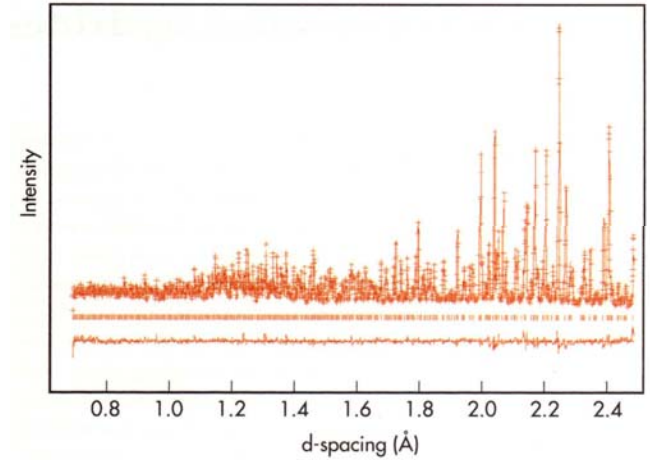
**Neutron scattering with isotope labeling** – **shapes and positions** of the Myosin Light Chain Kinase enzyme and calmodulin in the  $\text{Ca}^{2+}$ -calmodulin activated complex.

Use computational modeling based on crystallographic data to determine molecular shapes under various binding conditions

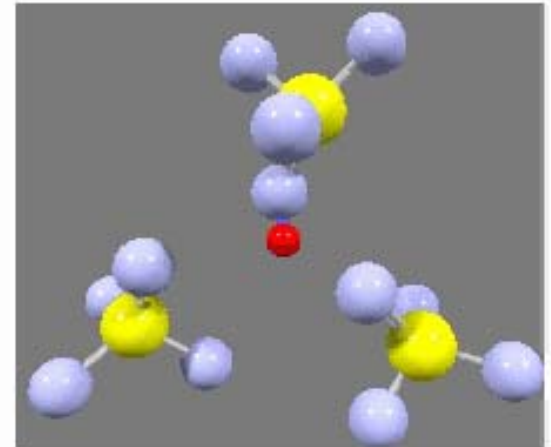
# Pictures & Movies are Today's Standard for Nano-Science Research



Today's Route



We need to provide images  
or movies

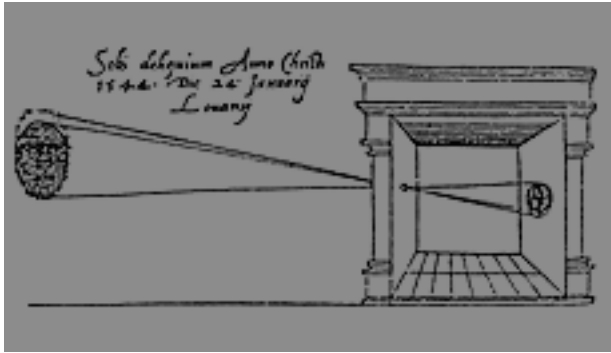


Reverse Monte Carlo of  $\text{CsDSO}_4$  fitted to diffraction data (McGreevy)

# What do we Need to do Better?

- Exploit complementarity of techniques
- Generate pictures not  $S(Q,E)$
- **Make better use of the neutrons we have**
  - Use the best known technology to optimize instrumentation
  - Develop better neutron focusing devices

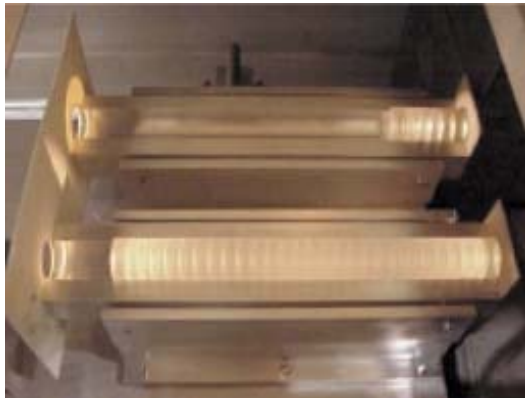
# Improved Neutron Optics



# Pin-hole Lens



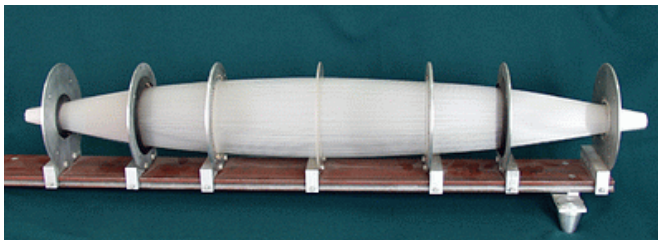
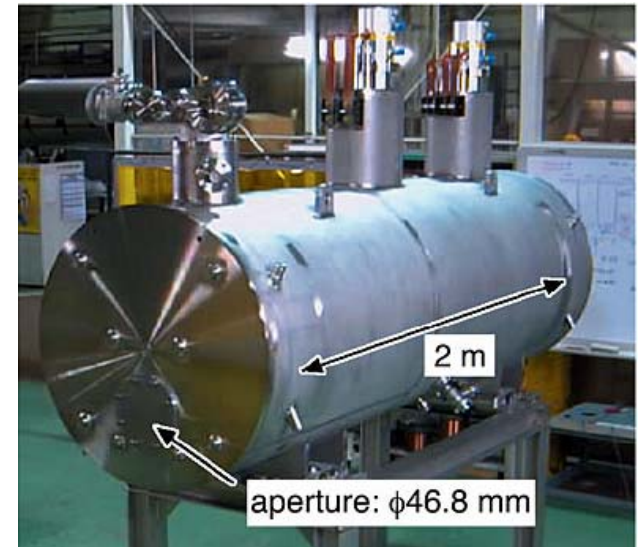
MgF<sub>2</sub>  
CRL  
at NIST



18 Å

8.4 Å

# Superconducting hexapole lens at RIKEN

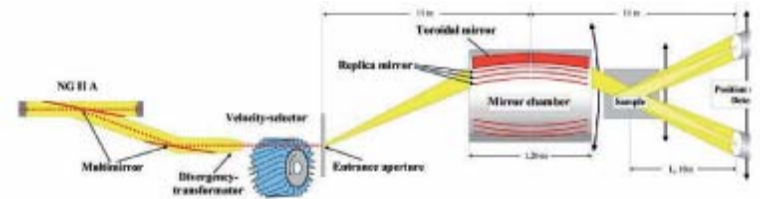


# Kumakhov lens

# Optical Elements Extend the Reach of Neutron Nano-Imagers



IN15 – ILL



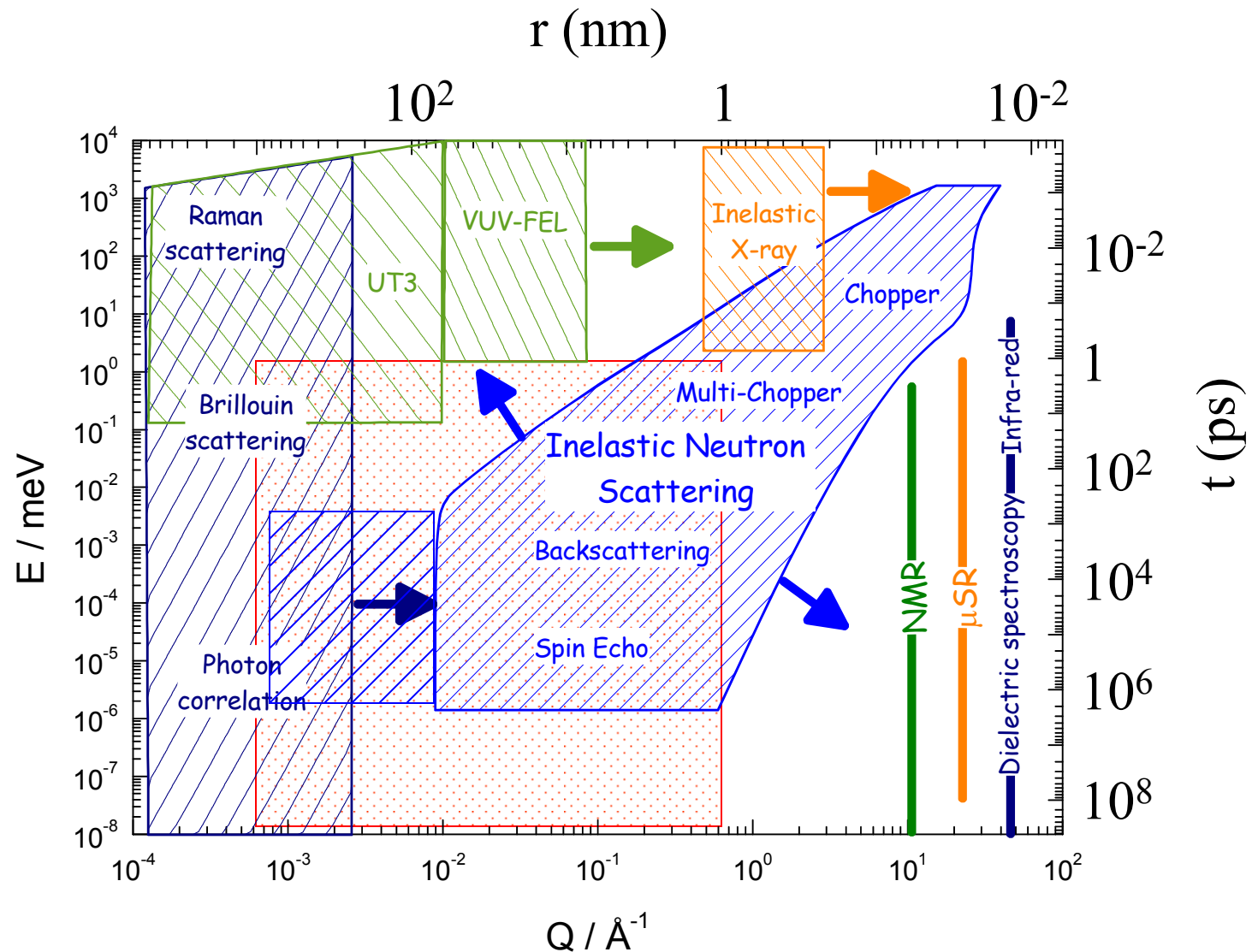
KWS-3 – Julich

Focusing torroidal mirrors provide higher intensity and allow smaller values of  $Q$  to be reached on SANS & neutron spin echo instruments

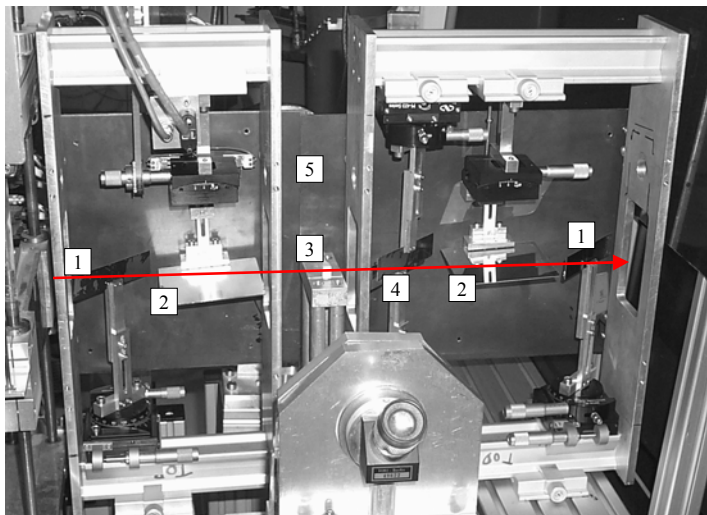
# What do we Need to do Better?

- Exploit complementarity of techniques
- Generate pictures & movies not  $S(Q,E)$
- Make better use of the neutrons we have
- **Design and build better neutron nanoscopes**
  - Extend accessible length and time scales
  - Allow nano-length-scales to be reached without loss of neutron intensity that arises from beam collimation, e.g by using the Neutron Spin Echo method
  - Etc (see session chaired by R. Gaehler)

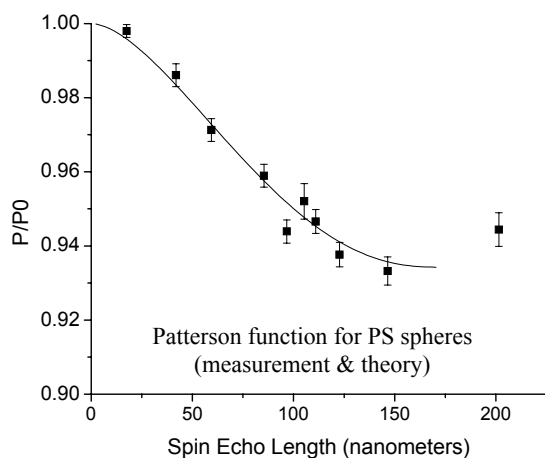
# Extension of the NSE Length-Scale Domain



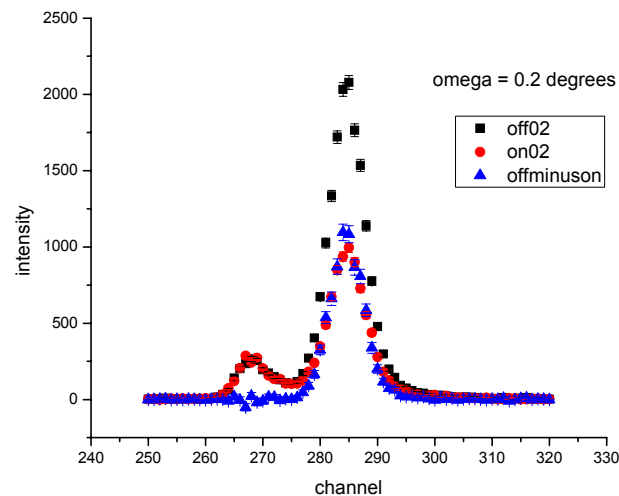
# High Angular Resolution Neutron Scattering without Beam Collimation



- Thin, magnetized  $\text{Ni}_{0.8}\text{Fe}_{0.2}$  films on silicon wafers (labelled 1, 2 & 4) are the principal physical components used for this new method.
- High angular resolution is obtained using Neutron Spin Echo.



A 200 nm correlation distance was achieved for SANS



Specular neutron reflection (blue) was separated from diffuse reflection with high fidelity. Black and red data include diffuse scattering

# What do we Need to do Better?

- Exploit complementarity of techniques
- Generate pictures & movies not  $S(Q,E)$
- Make better use of the neutrons we have
- Design and build better neutron nanoscopes
- Coordinated research effort on neutron instrumentation
  - Vision – a suite of neutron nanoscopes that probe the right length and time scales in weakly scattering samples
  - Possibility exists to optimize the SNS second target station & its instruments for nanoscience and biology if we start soon